

SOV/62-58-7-18/26

AUTHORS: ~~Shurkin, N. I.,~~ Timofeyeva, Ye. I., Dobrynina, T. P.,
Plotnikov, Yu. N., Dobryayeva, E. S., Kleymenova, V. M.

TITLE: The Reactions of N-Alkanes With a C_2-C_9 Structure in the
Presence of Alumino-Chromium-Potassium Catalysts
(Revrashcheniya n-alkanov sostava C_2-C_9 v prisutstvii
aljumokhromokaliyevogo katalizatora)

ORIGINAL: Izvestiya Akademii Nauk SSSR, Otdeleniye Khimicheskikh nauk,
1958, Nr 7, pp. 896-898 (USSR)

ABSTRACT: The production of alkenes and alkadienes by means of the
catalytic dehydration of the alkanes is of scientific and
practical interest. In the present brief report the authors
describe the reaction of n-alkanes (from pentane to nonane)
in the presence of alumino-chromium-potassium catalysts
of high activity and stability in the dehydration of iso-
pentane. It was shown that on the conditions assumed catalysts
could be obtained from these alkanes which contained 8-29 %
of unsaturated and 39-50 % of aromatic hydrocarbons. Finally
the authors point to the fact that after the dehydration of

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The Reactions of N-Alkanes With a C_7-C_9 Structure in the Presence of
Alumina-Chromium-Potassium Catalysts

the C_7-C_9 n-alkanes they obtained catalysts which contained
more than 14 % of alkenes. There are 1 figure and 5 references,
5 of which are Soviet.

ASSOCIATION: Institut organicheskoy khimii im. N. D. Zelinskogo Akademii
nauk SSSR
(Institute of Organic Chemistry imeni N. D. Zelinskiy, AS USSR)

SUBMITTED: February 17, 1956

SOV/62-58-8-16/22

AUTHORS: Shuykin, M. I., Cherkashin, M. I., Yakovlev, I. P.

TITLE: The Hydrolysis of Dicyclopentyl on a Skeleton Nickel-Aluminum Catalyst (Gidrogeneliz ditsiklopentila na skeletnom Ni - Al - katalizatore)

PERIODICAL: Izvestiya Akademii nauk SSSR, Otdeleniye khimicheskikh nauk, 1958, Nr 8, pp. 1008-1010 (USSR)

ABSTRACT: In the present short report the authors described their investigation of the reaction of the hydrolysis of dicyclopentyl on a skeleton nickel-aluminum catalyst at atmospheric pressure and at 200°. On these conditions the hydrolysis of only a five-membered ring with a simultaneous formation of products of the simple rupture of the C - C bonds of the five-membered ring as well as of alkyl cyclopentanes with a shortened side chain takes place. The scheme of the mechanism of the dicyclopentyl hydrolysis was devised and suggested by the authors. There are 1 table and 8 references, 6 of which are Soviet.

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The Hydrolysis of Dicyclopentyl on a Skeleton Nickel-Aluminium Catalyst

ASSOCIATION: Institut organicheskoy khimii im. N. D. Zelinskogo Akademii
nauk SSSR (Institute of Organic Chemistry imeni N. D. Zelinskiy,
AS USSR)

SUBMITTED: March 5, 1958

Card 2/2

AUTHORS: Shuykin, N. I., Pozdnyak, N. A., SOV/62-58-10-13/25
Yegorov, Yu. P.

TITLE: Catalytic Alkylation of Benzene by Alkene in the Vapor Phase
(Kataliticheskoye alkilirovaniye benzola alkenami v
parovoy faze) Communication 2: Benzene Alkylation With
3-Methyl Butene-1 (Soobshcheniye 2. Alkilirov-niye benzola
3-metilbutenom-1)

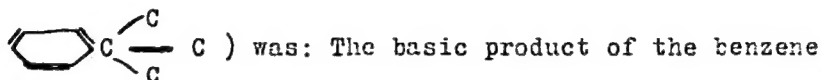
PERIODICAL: Izvestiya Akademii nauk SSSR. Otdeleniye khimicheskikh nauk,
1958, Nr 10, pp 1239 - 1244 (USSR)

ABSTRACT: The investigation of the reaction of the benzene alkylation
by 3-methyl butene-1 is the continuation of the
publications in this field of the benzene alkylation
in contact with zinc chloride, applied to aluminum oxide
(Refs 1-3). In the alkylation of benzene by 3-methyl
butene-1 carried out the formation of 2 amyl benzenes,
the 2-methyl-3-phenyl butane as well as small amounts
of 2-methyl-4-phenyl butane may be expected. From the
product of catalysis obtained amyl benzene (boiling point
189-189,5°; n_D^{20} 1,4929 and d_4^{20} 0,8736) was produced.

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Catalytic Alkylation of Benzene by Alkene in the Vapor Phase. Communication 2: Benzene Alkylation With 3-Methyl Butene-1 SOV/62-58-10-13/25

This substance was, however, neither similar to the 2-methyl-3-phenyl butane nor to 2-methyl-4-phenyl butane, but to the 2-methyl-2-phenyl butane. Its properties are: boiling point: 189-191°; n_D^{20} 1,4920 and d_4^{20} 0,8737. To explain this problem the spectra of the combination dispersion were used. The result of the investigation (in which the spectrum obtained was compared with the spectrum of the compound

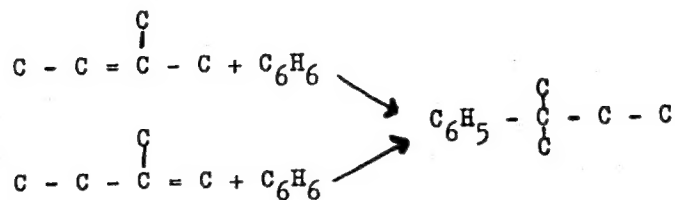
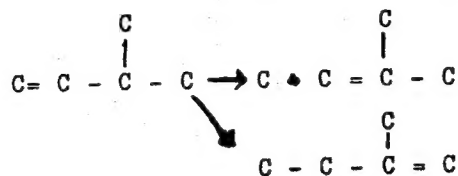


alkylation by 3-methyl butene-1 under the conditions assumed by the authors is: 2-methyl-2-phenyl butane. This hydrocarbon can be formed in the alkylation of benzene by 2-methyl butene-1 and 2-methyl butene-2. Based on the experimental data obtained the authors proposed the following reaction scheme:

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Catalytic Alkylation of Benzene by Alkene in the Vapor Phase. Communication 2: Benzene Alkylation With 3-Methyl Butene-1 SOV/62-58-10-13/25

SOV/62-58-10-13/25



There are 1 figure, 4 tables, and 7 references, 6 of which are Soviet.

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Catalytic Alkylation of Benzene by Alkene in the Vapor Phase. Communication 2: Benzene Alkylation With 3-Methyl Butene-1 SOV/62-58-10-13/25

ASSOCIATION: Institut organicheskoy khimii im. N.D.Zelinskogo Akademii nauk SSSR (Institute of Organic Chemistry imeni N.D. Zelinskiy AS USSR)

SUBMITTED: February 23, 1957

Card 4/4

5(3)

AUTHORS:

Bekauri, N.G., Shuykin, N.I.,
Yegorov, Yu.P.; ~~Shakarashvili~~, T.S.

SOV/62-58-11-17/26

TITLE:

Separation of Higher n-Alkanes From the Fraction With Its
Boiling Point at 190-350° of the Sokolovogorskaya and Mirzaani
Petroleums (Vydeleniye vysshikh n.alkanov iz fraktsii s t.kip.
190-350° sokolovogorskoy i mirzaanskoy neftey)

PERIODICAL:

Izvestiya Akademii nauk SSSR. Otdeleniye khimicheskikh nauk,
1958, Nr 11, pp 1376 - 1382 (USSR)

ABSTRACT:

In the present paper the authors investigated kerosene-gas oil
fractions of petroleum from the ~~Sokolov mountains~~ (Ural) and Mirzaani
(Gruzinskaya SSR) deposits. Under laboratory conditions fractions
were separated by means of direct distillation which evaporate in
the temperature range of 190-350°. The properties of the distillates
obtained are given (Table 1). 10 normal alkanes of the composition
 $C_{11}H_{24}$ - $C_{20}H_{42}$ from the fraction with its boiling point at 190-350°
were identified by means of physico-chemical methods and infrared
spectroscopy. The working methods applied were already described
in previous papers (Refs 1 - 6). In the investigated fractions
the content of each separated hydrocarbon was ascertained. The

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Separation of Higher n-Alkanes From the Fraction With SOV/62-58-11-17/26
Its Boiling Point at 190-350° of the Sokolovogorskaya and Mirzaani
Petroleums

data are given (Table 5). Parameters characterizing the motoric properties have been determined. The results of these determinations are given (Table 6). As can be seen from the table, the fraction with its boiling point at 190-350° of the Mirzaani petroleum as compared with the same fraction of the Sokolovogorskaya petroleum has a slightly lower characteristic. There are 2 figures, 6 tables, and 16 references, 11 of which are Soviet.

ASSOCIATION: Institut khimii im. P.G.Melikashvili Akademii nauk Gruz SSR
(Institute of Chemistry imeni P.G.Melikashvili, Academy of Sciences, Gruzinskaya SSR)
Institut organicheskoy khimii im.N.D. Zelinskogo Akademii nauk SSSR
(Institute of Organic Chemistry imeni N.D. Zelinskiy, Academy of Sciences, USSR)

SUBMITTED: March 22, 1957

Card 2/2

5(3)

SOV/62-58-12-12/22

AUTHORS: Shuykin, N. I., Tulupova, Ye. D., Polyakova, Z. P.

TITLE: ~~Contact~~-Catalytic Transformations of Metaxylol in the Presence of Alumosilicates (Kontaktno-kataliticheskiye prevrashcheniya metaksilola v prisutstvii alyumosilikatov)

PERIODICAL: Izvestiya Akademii nauk SSSR, Otdeleniye khimicheskikh nauk, 1958, Nr 12, pp 1476-1481 (USSR)

ABSTRACT: In the present paper the authors investigated the isomerization conditions of metaxylol in contact with "gumbrine" (Gruzinskaya SSR) and a synthetic alumosilicate catalyst as well as with aluminum oxide at different temperatures. The catalyst was supplied by the Ufimskiy neftepererabatyvayushchiy zavod (Ufa Works for Petroleum Processing). Furthermore, the same contacts containing smaller quantities of fine-disperse platinum (from 0.5 to 1%) were investigated. Isomerization takes place most easily with metaxylol in the presence of gumbrine at 450°, at an atmospheric pressure and a volume rate of 0.5.hours⁻¹. The yield of paraxylol under these conditions reaches 91.2% of the equilibrium composition. A decrease in pressure (50 torr) favors the complete removal of undesired reactions of methylation and demethylation, and

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Contact-Catalytic Transformations of Metaxylol in the Presence of Alumo-silicates

makes it possible to obtain up to 100% of the liquid catalyst with a paraxylol content of 15.6%. The use of hydrogen pressure (15 atmospheres) renders the reaction difficult owing to by-processes. The synthetic alumosilicate is less efficient than gumbrine, as it promotes by-processes and intensifies the formation of gas. In the presence of $\text{Pt-Al}_2\text{O}_3$, the metaxylol at 500° is also subjected to isomeric transformations in ortho- and para-isomers. Still toluene (up to 5.5%) and trimethyl benzenes (up to 2.5%) are formed in this connection. There are 2 figures, 2 tables, and 8 references, 3 of which are Soviet.

ASSOCIATION: Institut organicheskoy khimii imeni N. D. Zelinskogo Akademii nauk SSSR (Institute of Organic Chemistry imeni N. D. Zelinskiy, Academy of Sciences, USSR)

SUBMITTED: April 1, 1957

Card 2/2

BEKAURI, N.G.; SHUYKIN, N.I.; SHAKARASHVILI, T.S.; YEGOROV, Yu.P.

Normal alkanes included in the composition of liquid fuels for
jet engines and the synthesis of some of their analogs. Trudy
Inst.khim. AN Gruz.SSR 14:177-191 '58. (MIRA 13:4)
(Paraffins) (Jet planes--Fuel)

5(3)

SOV/55-58-2-30/35

AUTHORS:

Shuykin, N.I., Viktorova, Ye, A. and
Machel', G.

TITLE:

Hydrogenolysis of α -Methyl- α' -Ethyltetrahydrofurans With
a Nickel Aluminium Skeleton Catalyzer (Gidrogenoliz α -metil-
 α' -etil-tetragidrofurana na skeletnom nikel'-alyuminiye-
vom katalizatore)

PERIODICAL:

Vestnik Moskovskogo Universiteta. Seriya matematiki, mekhaniki,
astronomii, fiziki, khimii, V. 13, 1958, Nr 2, pp 217-222 (USSR)

ABSTRACT:

The paper starts from the former investigations of N.I. Shuykin [Ref 2,3] as well as of A.A. Ponomarev and V.V. Zelenkova [Ref 1]. The authors investigate the destructive hydrogenation of α -methyl- α' -ethylfurans and of its tetrahydroderivative in presence of a nickel aluminum skeleton catalyzer. Under 120° and 200° there takes place a hydrogenolysis of 42% and 75% respectively. By this example and some further ones it is shown that the tetrahydrofuran cycle is more stable than the furan cycle. The primary products of the hydro-genolysis are secondary beptylalcohols and probably hexanol - 3 too. There are 2 tables, 1 figure, and 9 references, 6 of which

Card 1/2

Hydrogenolysis of α -Methyl- α' -Ethyltetrahydrofurans SOV/55-58-2-30/35
With a Nickel Aluminum Skeleton Catalyzer

are Soviet, 1 is German, 1 English, and 1 French.

ASSOCIATION: Kafedra khimii nefiti (Chair of Petroleum Chemistry)

SUBMITTED: June 5, 1957

Card 2/2

23

5(2),5(3)

AUTHORS: Viktorova, Ye.A., Shuykin, M.I., Kirilenko, L.A., SOV/55-58-4-30/31
and Korostelava, G.S.

TITLE: Contact-Catalytic Change of the Phenoles. IV. Alkylation of
n-Cresol by Isoamylenes (Kontaktno-kataliticheskiye pravrash-
cheniya fenolov. IV. Alkilirovaniye n-kresola izoamilenami)

PERIODICAL: Vestnik Moskovskogo universiteta, Seriya matematiki, mekhaniki, astronomi-
i, 1958, No. 4, pp 231-234 (USSR)

ABSTRACT: The authors investigate the alkylation of n-cresol by 2-methyl-
butene-2, 2-methylbutene-1, and 2-methylbutene-3 in presence of
zinc chloride. The alkylation took place in the autoclave at 150°
and 2-3 atmospheres pressure. In all cases the authors obtained
4-methyl-2-tertiary-amylphenol; the corresponding isoamyl esters
of the n-cresol did not appear.
There is 1 table, and 5 references, 2 of which are Soviet, and
3 American.

ASSOCIATION: Kafedra khimii nefti (Chair of Petroleum Chemistry)

SUBMITTED: October 2, 1957

Card 1/1

SHUYKIN, N.I.; HEL'SKIY, I.F.

Catalytic dehydration of tetrahydrofuran homologues. Dokl. AN
Azerb. SSR 14 no.2:115-117 '58. (MIRA 11:4)

1. Institut organicheskoy khimii AN SSSR.
(Furan) (Dehydration (Chemistry))

SHUYKIN, N.I.; MINACHEV, Kh.M.; RYASHENTSEVA, M.A.

Producing aromatic hydrocarbons by dehydrogenation of narrow benzin fractions obtained in straight-run distillation. Dokl. AN Azerb. SSR 14:769-776 '58. (MIRA 11:11)

1. Institut organicheskoy khimii imeni N.D. Zelinskogo AN SSSR.
(Hydrocarbons) (Dehydrogenation)

BAKAURI, N.G.; SHUYKIN, N.I.

Contact catalytic conversion of normal tridecane and hexadecane
under pressure in a hydrogen atmosphere. Soob. AN Gruz. SSR 21
no. 4:421-428 0 '58. (MIRA 12:4)

1. AN GruzSSR, Institut khimii im. P.G. Melikishvili. Predstavleno
chlenom-korrespondentom Akademii G.V. TSitsishvili.
(Tridecane) (Hexadecane)

SHUYKIN, N. I.

79-1-4/53

AUTHORS: Nekrasova, V. A. , Shuykin, N. I. , Novikov, S. S.

TITLE: The Chlorination of Five- and Six-Membered Cyclanes
(Khlorirovaniye pyati- i shestichlennykh tsiklanov)

PERIODICAL: Zhurnal Obshchey Khimii, 1958, Vol. 28, Nr 1, pp.15-20 (USSR)

ABSTRACT: The chlorination processes of cycloparaffins had several times been the subject of investigations by many scientists which tried to find a way of conversion from the chemically inert cycloparaffins to their reactive halogen derivatives. The chlorination reactions of cyclo- and alkyl-paraffins are in many respects similar, but also show many a deviation from each other. Accurate results on the chlorination of these compounds, for example on the cyclohydrocarbons of mineral oil, are hitherto absent in publications. A detailed investigation of the chlorination conversions of the simplest cyclopentanes and cyclohexanes is not only of a high scientific interest, but also capable of indicating a new way for the synthesis of cyclic secondary alcohols of other derivatives as well as for the utilization of these synthetic hydrocarbons in tech-

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79-1-4/63

The Chlorination of Five- and Six-Membered Cyclanes

nology. The halide derivatives of cyclanes recently found a wide distribution as insecticides in agriculture. The thermal and photochemical chlorination of cyclopentane with dry and humid chlorine in the liquid and vapor phases were investigated. Conditions were found which permit to obtain mainly mono- or poly-chlorinated products as desired. The chlorination performed in the liquid phase has preference over all other chlorinations of five- and six-membered cyclanes. There are 5 tables, and 5 references, 3 of which are Slavic.

ASSOCIATION: Crimean Agricultural Institute and Institute for Organic Chemistry AN USSR
(Krymskiy sel'skokhozyaystvennyy institut i Institut organicheskoy khimii Akademii nauk SSSR)

SUBMITTED: January 7, 1957

AVAILABLE: Library of Congress

Card 2/2

1. Cyclic compounds
2. Cycloparaffins
3. Chlorination-Reaction
3. Chemistry

SHUYKIN, N. I.

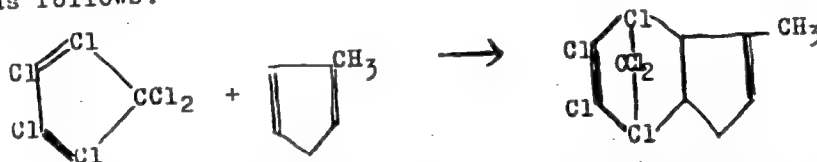
79-2-43/64

AUTHORS: Kukalenko, S. S., Mel'nikov, H. N.,
Naryshkina, T. I., Shuykin, N. I.

TITLE: Organic Insecticides and Fungicides (Iz oblasti
organicheskikh insektofungitsidov) XXXIII. Synthesis of Some
Derivatives of 4,7-Endomethylenetetrahydroindan (XXXIII. Sin-
tez nekotorykh proizvodnykh 4,7-endometilentetragidroindana).

PERIODICAL: Zhurnal Obshchey Khimii, 1958, Vol. 28, Nr 2, pp. 480-483 (USSR)

ABSTRACT: In order to investigate the insecticide-properties of chlor-
dane and heptachlorine homologues, an adduct was obtained from
hexachlorocyclopentadiene and 3-methylcyclopentadiene-2,4 by
heating at 85-105°C. It is assumed that the reaction takes place
as follows:



The product, a yellow viscous liquid, was chlorinated or bromiz-
ed resp. and the compounds obtained were tested for their in-

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79-2-43/64

Organic Insecticides and Fungicides. XXXIII. Synthesis
of Some Derivatives of 4,7-Endomethylenetetrahydroindan.

secticide-properties. It was found that all of them have a lower insecticide effect than "chlordane". These compounds have hitherto not been described in technical literature. The working methods and the specific data of the compounds are given. There are 5 references, 2 of which are Slavic.

ASSOCIATION: Scientific Institute for Fertilizers, Insecticides and Fungicides
and Institute for Organic Chemistry AS USSR (Nauchnyy institut po udobreniyam i insektofungitsidam i Institut organicheskoy khimii Akademii nauk SSSR).

SUBMITTED: January 16, 1957

AVAILABLE: Library of Congress

Card 2/2

SHUYKIN, N.I.; MINACHEV, Kh.M.; NOVIKOV, S.S.; KONONOV, N.F.; GARANIN, I.L.

Reforming straight-run gasolines by low-temperature dehydrogenation
on platinized charcoal. Zhur.prikl.khim. 31 no.11:1732-1738 N '58.
(MIRA 12:2)

(Gasoline)

SHUYKIN, N.I.; MINACHEV, Kh.M.; GARANIN, I.L.; NOVIKOV, S.S.; KONONOV, N.F.

Production of toluene concentrates from petroleum fractions by low-temperature dehydrogenation on platinated charcoal. Zhur.prikl.khim. (MIRA 12:2)

31 no.11:1765-1767 N '58.

(Toluene)

(Petroleum products)

(Dehydrogenation)

SOV/20-120-3-31/67

AUTHORS: Shurkin, N. I. Corresponding Member, Academy of Sciences,
USSR, Bel'skiy, I. P.

TITLE: Isomeric Transformation of γ -Oxides (Tetrahydrofuranes) of
Aliphatic Carbonyl Compounds (Izomerizatsiya γ -okisey (tetra-
gidrofuranov) v alifaticheskiye karbonil'nyye soedineniya)

PERIODICAL: Doklady Akademii nauk SSSR, 1958, Vol. 120, Nr 3, pp. 548-551
(USSR)

ABSTRACT: Several chemical transformations of alkylene-oxides are under
the action of various agents connected with an easily pro-
ceeding cleavage of the α -oxide cycle. The reaction mention-
ed in the title is among those well studied. It proceeds from
a heating to 300-500° or lower temperatures in the presence
of catalysts. Asymmetric α -oxides, which possess one or two
alkyl-substituents at one single carbon atom of the cycle,
by a cleavage of the cycle are isomerized preponderatingly
at that binding, which links the oxygen atom to that carry-
ing the substituent. This leads to the formation of aldehydes
(Ref 1). No analogous reaction was known to take place with
the compounds mentioned in the title. The catalysts, which

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Isomeric Transformation of γ -Oxides (Tetrahydrofuranes) of Aliphatic Carbonyl Compounds

are most active in the isomerization of α -oxides, with hydrofurane and its analogs lead to a dehydration reaction. It yields diene- and aromatic hydrocarbons. The authors performed the transformation reactions of tetrahydrofurane and of its α -substituents on platinated coal in the vapor phase at from 230-250°. They found that these substances under these conditions isomerize to aliphatic carbonyl compounds with a simultaneous cracking of the cycle at the C-O links.

Conclusions: 1) The α -oxide ring (tetrahydrofurane ring) is capable of the last mentioned transformations, by which aliphatic carbonyl compounds are formed. 2) If an alkyl radical (CH_3 , C_2H_5 , $n\text{-C}_3\text{H}_7$) is present in an α -position of the tetrahydrofurane cycle, the isomerization of the γ -oxide primarily (up to 90-95 %) takes place with a cracking of the cycle at the C-O link. This leads to the formation of corresponding aliphatic ketones. 3) The aldehydes, which are produced in small amounts in the isomerization of tetrahydrofurane and of α -alkyltetrahydrofuranes, suffer a cleavage at the C-O link 1,2 and are subject to a decarbonylation to the corresponding paraffin hydrocarbons under the prevailing reaction conditions.

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SOV/ 20-120-4-31/67
Isomeric Transformation of γ -oxides (Tetrahydrofuranes) of Aliphatic Carbonyl
Compounds

There are 2 tables and 7 references, 2 of which are Soviet.

ASSOCIATION: Institut organicheskoy khimii im. N. D. Zelinskogo Akademii
nauk SSSR
(Institute of Organic Chemistry imeni N. D. Zelinskij AS USSR)

SUBMITTED: January 11, 1958

1. Furan derivatives--Isomerism 2. Aliphatic compounds--Chemical
reactions 3. Carbonyls--Chemical reactions.

Card 3/4

00V/20-121-3-25/47

Academy of Sciences, USSR
Obrynia, U. S.

TITLE:

Reduction of *n*-Pentane and Isobutene in presence of
 mixed potassium catalysts (Degidrogenizatsiya
n-pentana i izobutena s prisutstviem smesnykh kalievogo
 katalizatorov)

1. 1958

Zhurnal khimicheskikh nauk SSSR, 1958, Vol. 121, Nr 3, pp.485-487
 (USSR)

2. 1958

Although mixed catalysts (Ref 4, 5) are mentioned in the title
 can be found already in earlier papers the authors of this
 paper succeeded for the first time in the determination of:
 a) the most favorable quantity ratio of the components and
 b) the conditions of the previous activation of the catalyst.
 These formulae are mentioned together with the formulae for
 regeneration. The influence of a) temperature, b) bulk velocity
 (ob'emyaya skorost') and c) the duration of the working
 cycles are investigated with respect to the capacity of de-
 hydrogenation of the mentioned catalyst. After each regeneration
 the catalyst was treated for 1,5 hours with hydrogen. a) Table 1

204 26-111-5-20/47

...the reaction of isopentane and Isopentane in presence of Aluno-Chromium-

are the results of the temperature experiments which guarantee the maximum yield of pentenes and isopentenes. 5) Table 2 presents the results of the experiments with the bulk velocity $W = 1.1 \text{ hours}^{-1}$. According to them the pentene and isopentene yields depend on the temperature. The lower it is, the greater the influence. Thus the change of velocity from 1.1 to 1.3 hours^{-1} reduces the isopentene yield from 36 to 27% and the yield of pentenes from 51 to 19%. At 550 and 570°C this influence practically ceases. 6) The higher the temperature the more rapidly decreases the activity of the catalyst with respect to time (Fig 1). During a working period of 18 minutes duration the isopentene content increases from 23.5% right to 45% in connection with a change of temperature from 500 to 550°C, computed for the isopentene having passed the catalyst. It is true that in a work period of 2 hours and 18 minutes and of 8 hours and 40 minutes the yields are somewhat higher than at the beginning of the work. At the end the main indices of the working catalyst, as well as the obtained products are shown under "Final" together with the obtained products.

NOV/20-121-5-25/47
Dehydrogenation of n-pentane and Isopentane in Presence of Alumo-Chromium-
Potassium Catalysts

the just mentioned conditions. There are 1 figure, 2 tables,
and 7 references, 4 of which are Soviet.

ASSOCIATION: Institut organicheskoy khimii im. N. D. Zelinskogo Akademii
nauk USSR
(Institute of Organic Chemistry imeni N. D. Zelinskogo, AS USSR)

SUBMITTED: January 4, 1958

Card 3/5

AUTHORS:

~~Shurkin, M. I.~~ Corresponding Member, SOV/20-122-4-24/57
Academy of Sciences, USSR, Bel'skiy, I. F.,
Karakhanov, R. A.

TITLE:

On the Reaction Mechanism of the Furane Cycle Catalytic
Hydrogenolysis (O mekhanizme reaktsii kataliticheskogo
gidrogenoliza furanovogo tsikla)

PERIODICAL:

Doklady Akademii nauk SSSR, 1958, Vol 122, Nr 4, pp 625 -
628 (USSR)

ABSTRACT:

The problem whether the reaction mentioned in the title
passes the hydration stage of double bonds of the cycle,
i.e. with a subsequent cleavage of the formed tetra-
hydrofurane cycle at one of the C-O-bonds, was raised
several times. This problem was rejected by several scien-
tists (Refs 1 - 3). It was proved that the tetra-hydro-
furane-cycle does not hydrogenolyze at all under the condi-
tions of the hydration in the liquid phase, whereas the
furane cycle hydrogenolyzes to a great extent. The authors
proved recently (Ref 6) that the homologs of tetra-hydro-
furane can be isomerized on Rt-coal at 250 - 300° in
aliphatic ketones. However, this reaction passes much more

Card 1/3

On the Reaction Mechanism of the Furane Cycle
Catalytic Hydrogenolysis

SOV/20-122-4-24/57

slowly than an immediate hydrogenolysis of the alkyl furanes themselves under the same conditions. Therefore, the tetrahydrofuranes cannot be considered to be intermediate products in the hydrogenolysis reaction of the furane compounds. All mentioned investigations led thus to the conclusion that the hydrogenolysis of the C-O-bond may take place either in the cycle of furane itself or in the dihydrofurane cycle. The formation of the latter as an intermediate product is confirmed by the transformation of silvane into γ -acetone-propylalcohol in the presence of water (Ref 7). In the present paper the authors investigated the hydration of the dihydrosilvane in the gaseous phase on platinized charcoal and on nickel to zinc oxide at 250 - 260°. Thus it was proved that not the hydrogenolysis of the C-O-bond, but the hydration of the C-C-bond in the cycle is the main reaction. The formation of methyl-propylketone in the hydration of the dihydrosilvane on Pt-C may take place in consequence of a secondary isomerization reaction of tetra-hydrosilvane. Tetra-hydrosilvane is not isomerized to methyl-propylketone on Ni-ZnO. The above results are interpreted differently

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On the Reaction Mechanism of the Furane Cycle
Catalytic Hydrogenolysis

SOV/20-122-4-24/57

by the authors. There are 13 references, 3 of which are
Soviet.

ASSOCIATION: Institut organicheskoy khimii im. N. D. Zelinskogo Akademii
nauk SSSR (Institute of Organic Chemistry imeni
N. D. Zelinskiy, AS USSR)

SUBMITTED: June 20, 1958

Card 3/3

CHUYKIN, N. I., MARYASHINA, T. I.

"Catalytic Synthesis of Cyclopentadiene Hydrocarbons."

Report submitted at the Fifth World Petroleum Congress, 30 May -
5 June 1959. New York.

S/081/61/000/019/037/085
B110/B101

AUTHORS: Shuykin, N. .. Viktorova, Ye. A.

TITLE: Contact catalytic transformation of cresols

PERIODICAL: Referativnyy zhurnal. Khimiya, no. 19, 1961, 157 - 158,
abstract 19Zh93 (Sb. "Novyye metody ratsional'n. ispol'zovaniya
mestn. topliv. Riga, AN LatvSSR, 1959, 265 - 276)

TEXT: A study has been made of the selective catalytic hydrogenation
(in a continuous laboratory apparatus) and alkylation with pentenes of
ortho-, meta-, and para-cresols (Ia-c). The following data are given:
initial I, hydrogenation temperature in °C, %Ni in the nickel/aluminum
oxide catalyst, yields of $C_6H_5CH_3$ (II), methyl cyclohexane (III),
p- $CH_3C_6H_4OH$, and p-methyl cyclohexanone in %: Ia, 175, 20, 1.6, 71, -,
-, Ic, 175, 20, traces, 43.33, -; Ib, 175, 20, 2, 34, 44, -; Ib, 125, 10,
-, -, 85.7, 2; Ib, 175, 10, 11, 41, 27, 3.5; Ib, 200, 10, 14, 25, -, -;
Ic, 175, 20, 2, 34, 44, -; Ic, 175, 10, 20, 52, 11, -; Ic, 175, 4, 5,
27.7, 54, -. 1,3,4-xlenols were hydrogenated on a 20% nickel/aluminum

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S/081/61/000/019/037/085
B110/B101

Contact catalytic transformation...

oxide catalyst. The following data are given: reaction temperature in °C, yields of II, III, o-(CH₃)₂C₆H₄, 1,2-dimethyl cyclohexane, 1,2-dimethyl-4-cyclohexanol, and 1,2-dimethyl 4-cyclohexanone: 150, traces, 1.8, 25, 29, 34.3, 7; 200, traces, 3.3, 17, 62.1, -, -, 250, 2.4, 22.3, 30.5, -, -. Reaction of Ib with (CH₃)₂C=CHCH₃ (IVa) or (CH₃)₂CHCH=CH₂ (IVb) (at 150°C and 2 - 3 atm on 20% ZnCl₂/80% Al₂O₃ catalyst, the amount of the catalyst used being 10% of the weight of the reagents) yielded 40% 3-CH₃-6-tert-C₅H₁₁Ar (V) (and some 3-CH₃-4-tert-C₅H₁₁Ar) (Ar = C₆H₃OH). One isomer only, 4-CH₃-2-tert-C₅H₁₁Ar (VI), was obtained in 40% yield from Ic with IVa, IVb, or CH₂=C(CH₃)C₂H₅. In all experiments, the initial iso-C₅H₁₀ dimerized, forming C₁₀H₂₀. Under the same reaction conditions, 1-pentene and 2-pentene yielded 70% alkyl cresols. Reaction of Ic with 1-pentene yielded 2-HO-3-CH₃C₆H₃CH(CH₃)C₃H₇ in 65% yield. The data given for the alkylation products (obtained with C₅H₁₀ and Ib or Ic) are b.p. in °C/mm Hg, n_D²⁰, d₄²⁰, m.p. in °C, and the m.p. of the

S/081/61/000/019/037/085
B110/B101

Contact catalytic transformation...

corresponding aryl glycolic acid in °C: V, 94 - 96/3, 1.5193, 0.9626, -,
142 - 143; VI, 125 - 125.5/3, -, -, 26.5 - 27, 126.5 - 127;
3-CH₃-6-CH(CH₃)(C₃H₇)Ar, 103 - 104/3, 1.5190, 0.9622, -, 98;
3-CH₃-6-CH(C₂H₅)₂Ar, 138 - 139/8, 1.5187, 0.9585, -, -, 3-CH₃-4-CH(C₂H₅)₂Ar,
147 - 148/8, 1.5148, 0.9693, -, 101. From Ib and piperylene (20% ZnCl₂ on
Al₂O₃), 70% of an alkylate was obtained, which consisted of
3-CH₃-6-CH(CH₃)-CH=CHCH₃Ar (VII) and 3-CH₃-4-CH(CH₃)CH=CHCH₃Ar (VIII) (90%),
and of 2,4,7-trimethyl chroman and 3,6-dimethyl-2-ethyl coumaran (10%).
Reaction of Ib with cyclopentadiene at 25 - 30 °C yielded the dimer of the
initial cyclopentadiene, and at 170 °C 2,3-cyclopentan-7-methyl-2,3-dihydro-
benzofuran (IX). With H₃PO₄ at 0 °C, 80% 3-methyl-6-(cyclopenten-2-yl)phenol
(X) and 3-methyl-4-(cyclopenten-2-yl)phenol (XI) were obtained. The
alkylation products obtained from Ib by treatment with piperylene are
characterized by their b.p. in °C/mm Hg, n_D²⁰, d₄²⁰, and by the m.p. of the
corresponding aryl glycolic acid in °C: VII, 136 - 137/9, 1.5312, 0.9847,

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S/081/61/000/019/037/085
B110/B101

Contact catalytic transformation...

108 - 108.5; VIII, 147 - 147.5/7, 1.5380, 0.9884, 118 - 118.5. The alkylation products obtained from Ib by treatment with cyclopentadiene are characterized by their b.p. in °C/mm Hg, n_D^{20} , d_4^{20} , m.p. in °C, and m.p. of the corresponding aryl glycolic acid in °C: 118 - 120/3, -, -, 69 - 70, 111 - 112; XI, 129 - 132/3, 1.5655, 1.0666, -, 129; 3-methyl-6-cyclopentyl phenol, 113 - 115/3, 1.5495, 1.0087, -, 101 - 102; 3-methyl-4-cyclopentyl phenol, 123 - 125/3, 1.5549, 1.0498, -, 101 - 107; IX, 138 - 140, 1.5474, 1.0608, -, -. [Abstracter's note: Complete translation.]

Card 4/4

5(7)
AUTHORS:

SOV/62-59-1-18/33
Shuykin, N. I., Bekauri, N. G., Shakarashvili, T. S.

TITLE:

Contact-Catalytic Transformations of n-Hexadecane in the Presence of Gumbrine (Kontakno-kataliticheskiye pre-vrashcheniya n. geksadekana v prisutstvii gumbrina)

PERIODICAL:

Izvestiya Akademii nauk SSSR. Otdeleniye khimicheskikh nauk, 1959, Nr 1, pp 110 - 114 (USSR)

ABSTRACT:

In the present paper the authors investigated the transformation of n-hexadecane in the presence of natural and activated gumbrine. It was shown for the first time that these transformations at high temperatures (450 and 400°) and hydrogen pressure of 30 atmospheres absolute pressure in contact with natural and activated gumbrine take place almost equally. In this case the degree of transformation of n-hexadecane amounts to 58%. The effect exercised by activated gumbrine saturated with zinc chloride solution (20%) differs, however, considerably from that of pure gumbrine. In this case the formation of cracking gases is reduced approximately a threefold and the yield of the readily boiling fraction (up to 100°), which mainly consists

Card 1/2

Contact-Catalytic Transformations of n-Hexadecane in the Presence of Gumbrine SOV/62-59-1-18/33

of isomer alkanes, strongly increases. Thus n-hexadecane is transformed in a degree of 80%. Natural gumbrine was supplied by pit Nr 1 "Tetra" (30 m deep) of the deposit near Gumbri Gruzinskaya SSR. The compositions and properties of natural and activated gumbrine are given in table 1. The main reaction during the transformation of n-hexadecane is its hydro-cracking in which isomerization products are formed. The dehydrocyclization takes place simultaneously in which high-boiling aromatic hydrocarbons are formed. There are 1 figure, 6 tables, and 7 references, 5 of which are Soviet.

ASSOCIATION:

Institut organicheskoy khimii im. N. D. Zelinskogo Akademii nauk SSSR (Institute of Organic Chemistry imeni N. D. Zelinskiy of the Academy of Sciences, USSR) Institut khimii im. P. G. Melikishvili AN GruzSSR (Institute of Chemistry imeni P. G. Melikishvili, AC Georgian SSR)

SUBMITTED:

May 17, 1957

Card 2/2

SOV/62-59-1-30/38

5(3)

AUTHORS:

Shuykin, N. I., Cherkashin, N. I.

TITLE:

Hydrogenolysis of Hydrocarbons of the Pentamethylene Series
on the Ni-Al Skeleton Catalyst (Gidrogenoliz uglevodorodov
pentametilenovogo ryada na skeletnom Ni-Al katalizatore)

PERIODICAL:

Izvestiya Akademii nauk SSSR. Otdeleniye khimicheskikh nauk,
1959, Nr 1, pp 168 - 170 (USSR)

ABSTRACT:

In the preceding paper (Ref 1) the authors have shown that
pentamethylene hydrocarbons in the presence of a Ni-Al
skeleton catalyst undergo hydrogenolysis. In
addition to the products of a simple opening of C-C bonds
of the 5-membered cycle, hydrocarbons with shortened carbon
chain are formed there. In the present paper cyclopentane,
methyl cyclopentane and ethyl cyclopentane were investigated
in this direction. It was found that the above lowest
homologs of cyclopentane are subject to the same laws. It
was stated that alkanes with shortened chain are formed at
the expense of the carbon atoms contained in the penta-
methylene cycle. It was demonstrated that cyclopentane is

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Hydrogenolysis of Hydrocarbons of the Pentamethylene
Series on the Ni-Al Skeleton Catalyst

SOV/62-59-1-30/38

opened by 51.5% at a single flow at 200°. Under equal conditions, methyl cyclopentane and ethyl cyclopentane are opened accordingly by 43 and 40%. Under these conditions, the isoalkanes formed are partly hydrogenated into gaseous hydrocarbons in a destructive way. There are 3 tables and 1 Soviet reference.

ASSOCIATION:

Institut organicheskoy khimii im. N. D. Zelinskogo Akademii
nauk SSSR (Institute of Organic Chemistry imeni N. D. Ze-
linskiy of the Academy of Sciences, USSR)

SUBMITTED:

June 12, 1958

Card 2/2

SOV/62-59-2-18/40

5(3)

AUTHORS:

Shuykin, N. I., Pozdnyak, N. A.

TITLE:

Catalytic Alkylation of Benzene by Alkenes in the Vapor Phase
(Kataliticheskoye alkilirovaniye benzola alkenami v parovoy
faze). Communication 3. Alkylation of Benzene by Pentene-1
(Soobshcheniye 3. Alkilirovaniye benzola pentenom-1)

PERIODICAL:

Izvestiya Akademii nauk SSSR. Otdeleniye khimicheskikh nauk,
1959, Nr 2, pp 304-307 (USSR)

ABSTRACT:

In the present paper benzene was alkylated with pentene-1 at 350° and a pressure of 40 atmospheres in contact with zinc chloride (30%) on aluminum oxide. Instead of the 2-phenyl pentane (I) and n-amyl benzene (II) expected, by means of the spectrum analysis 2-methyl-2-phenyl butane (III) and 3-methyl-4-phenyl butane (IV) or 2-methyl-3-phenyl butane (V) were found. The amyl benzenes mentioned can be formed on alkylation of benzene with 2-methyl butene-2 or with 2-methyl butene-1. These latter were obtained, under assumed conditions, by means of isomerization of pentene-1. In the fractions of the catalysts which have a boiling point above 210°, α- and β-methyl naphthalene was detected by means of Raman-spectra. The latter is

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SOV/62-59-2-18/40
Catalytic Alkylation of Benzene by Alkenes in the Vapor Phase. Communica-
tion 3. Alkylation of Benzene by Pentene-1

formed apparently in consequence of the dehydrocyclization of
3-methyl-4-phenyl butane. As to the formation of α -methyl
naphthalene it might be assumed to be the isomerization prod-
uct of β -methyl naphthalene. The authors expressed their
gratitude to Yu. P. Yegorov for the recording of the Raman-
spectra. There are 2 tables and 3 references, 2 of which are
Soviet.

ASSOCIATION:

Institut organicheskoy khimii im. N. D. Zelinskogo Akademii
nauk SSSR (Institute of Organic Chemistry imeni N. D.
Zelinskiy of the Academy of Sciences, USSR)

SUBMITTED:

May 20, 1957

Card 2/2

5(3)

AUTHORS:

TITLE:

PERIODICAL:

ABSTRACT:

Card 1/2

SOV/62-59-2-19/40

Shuykin, N. I., Berdnikova, N. G., Kashkovskaya, L. K.

Transformations of Individual Xylenes in Presence of a Nickel-alumina Catalyst at Hydrogen Pressure (Prevrashcheniya individual'nykh ksilolov v prisutstvii nikel'-glinozemnogo katalizatora pod davleniyem vodoroda)

Izvestiya Akademii nauk SSSR, Otdeleniye khimicheskikh nauk, 1959, Nr 2, pp 308-313 (USSR)

In the investigation of the behavior of some aromatic hydrocarbons in the presence of platinum and nickel-aluminum oxide catalysts it could be proved (Refs 6,7) that they undergo a number of fundamental transformations at high temperatures and hydrogen pressure. In order to continue these observations and to find the possibility of obtaining toluene and benzene from xylenes, the behavior of individual m-, p-, and o-xylenes was investigated in the present paper. The studies were carried out at temperatures of from 300 to 460°, at hydrogen pressure of 25 and 50 atmospheres in the presence of the nickel-aluminum oxide catalyst with a nickel content of 10, 20, and 30%. It was found that the principal reaction in the catalysis of isomeric

SOV/62-59-2-19/40
Transformations of Individual Xylenes in Presence of a Nickel-alumina
Catalyst at Hydrogen Pressure

xylenes is the demethylation of the initial products. Toluene and benzene are formed in this connection. At temperatures of 300 - 400° also the hydrogenation of the benzene ring was observed where dimethyl cyclohexane, methyl cyclohexane and cyclohexane were formed. Device and methods applied to this investigation have been described previously (Refs 9,10). There are 4 tables and 10 references, 7 of which are Soviet.

ASSOCIATION:

Institut organicheskoy khimii im. N. D. Zelinskogo Akademii nauk SSSR (Institute of Organic Chemistry imeni N. D. Zelinskiy of the Academy of Sciences, USSR)

SUBMITTED:

May 25, 1957

Card 2/2

MAKARENKO, V.V.; MESHCHERYAKOV, A.P.; PANCHENKOV, G.M.; PLATE, A.F.;
SHUYKIN, N.I.; YAKOVLEVSKIY, V.V.

Effect of the structure of individual hydrocarbons and ethers on
their combustion rate. Izv. vys. ucheb. zav.; neft' i gaz 2 no.4:
71-78 '59. (MIRA 12:10)

1. Moskovskiy institut neftekhimicheskoy i gazovoy promyshlennosti
im. akad. I.M. Gubkina.
(Hydrocarbons) (Ethers) (Combustion)

5(3)

AUTHORS:

Shuykin, N. I., Cherkashin, M. I.

SOV/62-59-3-18/37

TITLE:

Catalytic Transformations of 1-Methyl-4-isopropylcyclohexane in Conditions of High Temperature and Hydrogen Pressure (Kataliticheskiye prevrashcheniya 1-metil-4-izopropiltsiklogeksana v usloviyakh povyshennykh temperatury i davleniya vodoroda)

PERIODICAL:

Izvestiya Akademii nauk SSSR. Otdeleniye khimicheskikh nauk, 1959, Nr 3, pp 507-510 (USSR)

ABSTRACT:

It was proved (Ref 1) with respect to the effect of alkyl groups in the cyclohexane ring that methylcyclohexane is dehydrogenated in a higher degree than cyclohexane and ethylcyclohexane. In this connection the action of two substituents on the behavior of the cyclohexane ring under platforming conditions was investigated here. 1-methyl-4-isopropylcyclohexane was investigated in an apparatus with continuous flow at 450° and a pressure of 20 atmospheres in the presence of 0.5 % Pt on Al₂O₃ and 0.5 % Pt on ZrO₂. The properties of catalyzates and the gas composition are given in table 1. The composition of the catalyzates according to fractions are presented in tables 2 and 3. From these tables it may be seen that during

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Catalytic Transformations of 1-Methyl-4-isopropyl-
cyclohexane in Conditions of High Temperature and Hydrogen Pressure

SOV/62-59-3-18/37

the transformations of 1-methyl-4-isopropylcyclohexane on platinum-aluminum oxide the principal reactions are the dehydrogenation and dealkylation. The principal mass of the catalyzate consisted of toluene, 1-methyl-4-isopropylbenzene, and a mixture of dimethylethylbenzenes. The naphthene-paraffin portion contained also methylcyclohexane, 1,3-dimethylcyclopentane and a certain amount of unchanged 1-methyl-4-isopropylcyclohexane. On platinum-circonium oxide the principal reaction was the dehydrogenation of 1-methyl-4-isopropylcyclohexane into 1-methyl-4-isopropylbenzene. The formation of toluene and methylcyclohexane was negligible. It was worthy of note that naphthalene was found in the catalyzates. The occurrence of a considerable quantity of methylcyclohexane (5 %) and toluene (25 %) in the catalyzate obtained on Pt-Al₂O₃ permits the assumption that the dealkylation of the initial product 1-methyl-4-isopropylcyclohexane takes place prior to its aromatization. Only an insignificant compression of the methylcyclohexane ring takes place there,

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Catalytic Transformations of 1-Methyl-4-isopropyl-
cyclohexane in Conditions of High Temperature and Hydrogen Pressure

SOV/62-59-3-18/37

1,3-dimethylcyclopentane being formed. There are 3 tables
and 3 references, 2 of which are Soviet.

ASSOCIATION:

Institut organicheskoy khimii im. N. D. Zelinskogo Akademii
nauk SSSR (Institute of Organic Chemistry imeni N. D. Zelin-
skiy of the Academy of Sciences, USSR)

SUBMITTED:

July 6, 1957

Card 3/3

SOV/62-59-4-22/42

5(3)

AUTHORS:

Romadan, I. A., Grikit, E. Ya.,
Shuykin, N. I.

TITLE:

Alkylation of Toluene by Molecular Compounds of Alcohols With
Boron Fluoride Under Pressure (Alkilirovaniye toluola molekul-
yarnymi soyedineniyami spirtov s ftoristym borom pod
davleniyem)

PERIODICAL:

Izvestiya Akademii nauk SSSR. Otdeleniye khimicheskikh nauk,
1959, Nr 4, pp 705-709 (USSR)

ABSTRACT:

In the present paper toluene was alkylated in the autoclave
with n-propyl, isopropyl, n-butyl, isobutyl, isoamyl, and
n-hexyl alcohol in the presence of BF_3 at 170-180° and under
40-60 atmospheres. As a result 1,4-dialkyl- and 1,2,4-trialkyl
benzenes were obtained in a 62-87 % yield of initial toluene.
1-methyl-4-alkyl benzenes amounted to 53-78 % of the total
quantity of the alkyl benzenes obtained, whereas the yield in
1-methyl-2,4-dialkyl benzenes was not more than 9-17 %. The
monoalkyl toluenes precipitated from the catalysates had con-
stants similar to the data for synthetic alkyl toluenes. Upon
alkylation of toluene with n-propyl alcohol,

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SOV/62-59-4-22, '42

Alkylation of Toluene by Molecular Compounds of
Alcohols With Boron Fluoride Under Pressure

1-methyl-4-isopropyl benzene was obtained: melting point 177.4° ,
 n_D^{20} 1.4915, d_4^{20} 0.8575 as compared to melting point 177° ,
 n_D^{20} 1.4909, d_4^{20} 0.8573 (Ref 7). Upon alkylation with isopropyl
alcohol, 1-methyl-4-isopropyl benzene was obtained: melting
point 177° , n_D^{20} 1.4911, d_4^{20} 0.8573 as compared to melting point
 177.25° , n_D^{20} 1.4909, d_4^{20} 0.8573 (Ref 7). The constants of other
hydrocarbons obtained are shown in table 1. The experimental
data show that the yield in alkyl toluenes depends on the
molar ratio of the initial reagents. At a molar ratio of
toluene : alcohol = 1 : 1 the yield in alkyl toluenes was
about 20-25 % lower than at a ratio of 1 : 2 (Table 2). In
addition to the mentioned monoalkyl toluenes, disubstituted
alkyl toluenes were obtained, as, e.g. 1-methyl-2,4-diiso-
propyl-, 2,4-di-n-butyl; and 1-methyl-2,4-di-n-propyl-,
1-methyl-, 1-methyl-2,4-diisobutyl-, and 1-methyl-2,4-diiso-
amyl benzenes

Card 2/3

Alkylation of Toluene by Molecular Compounds of
Alcohols With Boron Fluoride Under Pressure

SOV/62-59-4-22/42

which were not yet described (Table 1). There are 2 tables
and 12 references, 7 of which are Soviet.

ASSOCIATION:

Latviyskiy gosudarstvennyy universitet (Latviya State Uni-
versity). Institut organicheskoy khimii im. N. D. Zelinskogo
Akademii nauk SSSR (Institute of Organic Chemistry imeni
N. D. Zelinskiy of the Academy of Sciences, USSR)

SUBMITTED:

July 9, 1957

Card 3/3

SOV/62-59-4-23/42

5(3)

AUTHORS:

Bel'chev, F. V., Shuykin, M. I.,
Novikov, S. S.

TITLE:

Catalytic Synthesis of Aliphatic Amines Over Mixed Oxidizing Catalysts Under Increased Pressure (Kataliticheskiy sintez alifaticheskikh aminov na smeshannykh okisnykh katalizatorakh pri povyshennom davlenii)

PERIODICAL:

Izvestiya Akademii nauk SSSR. Otdeleniye khimicheskikh nauk, 1959, Nr 4, pp 710-714 (USSR)

ABSTRACT:

The experimental results found in the present work show that a considerable yield of amines can be obtained by a scientifically founded selection of catalysts and conditions of aminating alcohols. The relative activity of the following catalysts was investigated: pure aluminum oxide (85 %) + ferrous oxide (15 %), aluminum oxide (95 %) + titanium oxide (5 %), aluminum oxide (90 %) + magnesium oxide (10 %) and industrially produced aluminum silicate. The alcohols were twice distilled before the investigation was carried out. Their properties were in good agreement with those described in technical publications (Refs 4-6). The experiments were carried out in a continuous flow apparatus (Fig). The experimental results

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Catalytic Synthesis of Aliphatic Amines Over Mixed
Oxidizing Catalysts Under Increased Pressure

SOV/62-59-4-23/42

are shown in tables 1, 2, and 3. The optimum conditions for the catalytic amination of n-butanol were determined on an aluminum-titanium catalyst which showed the highest activity: ammonia pressure 8.5 atmospheres, reaction rate 0.37 h^{-1} and 370° . A further pressure increase reduces the amine yield, probably because of the condensation of ammonia under the conditions assumed. With catalytic amination of alcohols less gas formation is observed, probably because of the slower process of alcohol decomposition. If the temperature exceeds the optimum value the gas formation is increased and amine yield reduced. It was observed that amination stops upon deviation from the optimum passage rate of the alcohol and ammonia vapors over the catalyst. As compared to pure aluminum oxide, almost all mixed catalysts developed a strong activity (Tables 2 and 3). Under the conditions assumed small quantities of unsaturated hydrocarbons, hydrogen, paraffin hydrocarbons, aldehydes, and nitriles were formed, in addition to the amines, on all catalysts investigated. The properties of the amines separated out of the catalysates are shown in

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Catalytic Synthesis of Aliphatic Amines Over Mixed
Oxidizing Catalysts Under Increased Pressure

SOV/62-59-4-23/42

table 4. There are 1 figure, 4 tables, and 26 references,
4 of which are Soviet.

ASSOCIATION:

Belorusskaya sel'skokhozyaystvennaya akademiya (Belorussian
Agricultural Academy). Institut organicheskoy khimii in.
N. D. Zelinskogo Akademii nauk SSSR (Institute of Organic
Chemistry imeni N. D. Zelinskiy of the Academy of Sciences,
USSR)

SUBMITTED:

July 12, 1957

Card 3/3

5 (3)

AUTHORS:

Tinofareva, Ye. A., Shuykin, N. I.,
Dobrynina, T. P., Kleynenova, V. K.

307/62-59-5-16/40

TITLE:

Effect of **Space Velocity** on the Catalytic Dehydrogenation
of n-Pentane and Isopentane (Vliyaniye ob'yemnoy skorosti na
kataliticheskoye degidrirovaniye n.pentana i isopentana)

PERIODICAL:

Izvestiya Akademii nauk SSSR. Otdeleniye khimicheskikh nauk,
1959, Nr 5, pp 875-879 (USSR)

ABSTRACT:

In previous works (Refs 1-3) on the temperature effect on the
dehydrogenation reaction of n-pentane and isopentane the authors
showed that this reaction attains equilibrium at a **space veloc-
ity of 0.5/hr** in the temperature range between 500 and 550°. **ity of 0.5/hr**
in connection with it the effect of the throughput rate on
the reaction mentioned has been investigated in this work.
n-Pentane was investigated in the temperature range between
500-550°, isopentane in the same temperature range and at
575°. The **space velocity** was varied from 0.3-1.7/hr. The
method of analysis and the investigation conditions were
similar to those of the works (Refs 2, 3). The results
obtained are shown in tables 1-2 and figures 1 and 2. Tables 1
and 3 show the effect of the **space velocity** on the dehydro-

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SCV/62-59-5-16/10

Effect of **Space Velocity** on the Catalytic
Dehydrogenation of n-Pentane and Isopentane

generation reaction of n-pentane and isopentane, respectively. Tables 2 and 4 show the molar balance of the reaction and the selectivity of the catalyst of the two compounds mentioned. The figures show the molar balance depending on the throughput rate. Aluminum chromium potassium catalysts were used in the reactions. It appeared that the dependence of the mono-olefin yield on the **space velocity** decreases with temperature rise. If the reaction is carried out at a **space velocity** of 0.3-1.1/hr at 550 and 575° the yield is not changed at all. Optimum reaction conditions prevail at 550° and a **space velocity** of 1.1/hr. Under these conditions 45 mole % isopentene and 5 mole % isoprene were formed from isopentane. 39 mole % pentene are formed from n-pentane. The isoprene yield is practically independent of the change of **space velocity**; it never surpasses 5-6 mole % of the isopentane let through. There are 2 figures, 4 tables, and 3 Soviet references.

Card 2/3

Effect of **Space Velocity** on the Catalytic
Dehydrogenation of n-Pentane and Isopentane

SOV/62-59-5-16/40

ASSOCIATION: Institut organicheskoy khimii im. N. D. Zelinskogo Akademii
SSSR (Institute of Organic Chemistry imeni N. D.
Zelinskoy of the Academy of Sciences, USSR)

SUBMITTED: July 12, 1957

Doc# 173

5 (3,4)
AUTHORS:

SOV/62-59-6-21/36
Shuykin, N. I., Pozdnyak, N. A.

TITLE:

Catalytic Transformations of Pentene-1 and 3-Methylbutene-1
(Kataliticheskiye prevrashcheniya pentena-1 i 3-metilbutena-1)

PERIODICAL:

Izvestiya Akademii nauk SSSR. Otdeleniye khimicheskikh nauk,
1959, Nr 6, pp 1088 - 1090 (USSR)

ABSTRACT:

As is well known from publications (Refs 1-10,11,12, the latter two are previous papers by the authors) the isomeric transformation (ramification, transposition of the double bonds) by the influence of different catalysts takes place in a different way and to a different degree. In this connection the present investigation dealt with the catalytic transformation of the substances mentioned in the title with zinc chloride on saturated aluminum oxide at a pressure of 40 atmospheres and a temperature of 350°. The investigations were carried out in a continuously working apparatus. They showed that with the given conditions the compounds mentioned readily isomerize to 2-methylbutene-1 and 2-methylbutene-2. The isopentanes were hydrated by transposition of the hydrogen, and transformed into methylbutene. Part of the pentenes is also polymerized under formation of dimers. In the experimental part the catalytic transformation

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Catalytic Transformations of Pentene-1 and 3-Methylbutene-1

SOV/62-59-6-21/36

of both substances is described in detail. The two tables give the results of the distillation of the catalysates (I,II) of both substances. The catalysate (I) contained 31.5% 2-methylbutene, 6.8% 3-methylbutene-1, 17.5% 2-methylbutene-2, and 6.7% 2-methylbutene-1. The other products were polymerization products. The catalysate (II) contained 2-methylbutene 32%, isopentene 40.8%, and polymerization products 27.2%. There are 1 table and 14 references, 7 of which are Soviet.

ASSOCIATION: Institut organicheskoy khimii im. N. D. Zelinskogo Akademii nauk SSSR (Institute of Organic Chemistry imeni N. D. Zelinskiy of the Academy of Sciences, USSR)

SUBMITTED: September 27, 1957

Card 2/2

5 (3)
AUTHORS:

Shuykin, N. I., Brusnikina, V. M.

SOV/62-59-7-12/38

TITLE:

Hydration of Pyridine and of α -Picoline on a Nickel-aluminum
Skeleton Catalyst (Gidrirovaniye piridina i α -pikolina na
skeletnom nikel'-alyuminiyevom katalizatore)

PERIODICAL:

Izvestiya Akademii nauk SSSR. Otdeleniye khimicheskikh nauk,
1959, Nr 7, pp 1288 - 1293 (USSR)

ABSTRACT:

Initially it is reported on the different methods of hydration of pyridine existing at present and their results. Sadikov and Mikhaylov (Ref 4), Ushakov (Ref 5) and Tulupov (Ref 6) are mentioned of the Russian research workers. The catalytic hydration process of pyridine is a process influenced by several factors. In the present paper the authors investigate the hydration of pyridine and its next homologue, α -picoline, in a continuously operating apparatus on a nickel skeleton catalyst at 140 and 200°. It was found that not only piperidine, but also α -picoline, β -methylpyrrene, α -propylpiperidine, as well as N-cyclopentylpiperidine are formed during the catalytic process. The hydration of α -picoline under equal conditions leads mainly to the formation of α -pipecoline and β -methylpyrrene. The activity of the catalyst during the transformation process was determined.

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Hydration of Pyridine and of α -Picoline on a
Nickel-aluminum Skeleton Catalyst

SOV/62-59-7-19/38

from the change of the refractive index of pyridine. Table 1 gives the conditions of the hydrations in the two experiments carried out. The catalysate was fractionated in a column with 42 theoretical bottoms. Fractions of the individual substances or azeotropic mixtures were obtained. The properties of the catalysate fractions are given in tables 2 and 3. The hydration conditions and the properties of the fractions of the catalysate of α -picoline at 140 and 200° are given in tables 4, 5, and 6. There are 6 tables and 15 references, 7 of which are Soviet.

ASSOCIATION: Institut organicheskoy khimii im. N. D. Zelinskogo Akademii nauk
SSSR (Institute of Organic Chemistry imeni N. D. Zelinskiy of
the Academy of Sciences, USSR)

SUBMITTED: November 18, 1957

Card 2/2

5 (3), 5 (4)
AUTHORS:

Timofeyeva, Ye. A., Shuykin, N. I.,
Dobrynina, T. P., Kleymenova, V. M.

SOV/62-59-9-16/40

TITLE:

Lifetime of a Catalyst Without Regeneration at the Catalytic
Dehydrogenation of Isopentane

PERIODICAL:

Izvestiya Akademii nauk SSSR. Otdeleniye khimicheskikh nauk,
1959, Nr 9, pp 1623 - 1626 (USSR)

ABSTRACT:

The present article is the continuation of the investigations of these authors on the dehydrogenization reaction of pentanes on aluminum chromium potassium catalysts. The reaction scheme is described. This showed that the yield of amylenes at higher reaction temperature is independent in a wide range of the volume rate of the initial substance. In the present paper investigation was carried out on the influence on the dehydrogenation reaction of the duration of the operation cycle of the catalyst between regenerations at 500, 527, and 550°. At 500 and 550°, 28 experiments were carried out until the regeneration of the catalyst, and at 527°, 33 experiments. Table 1 contains the indices of the different work cycles. It showed that when the reaction temperature was higher the activity of the catalyst decreased faster. The maximum yield was obtained at 500° in ex-

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Lifetime of a Catalyst Without Regeneration at the
Catalytic Dehydrogenation of Isopentane

SOV/62-59-9-16/40

periment Nr 8. At 527 and 550° the maximum yield was obtained already at experiment Nr 3. The yield was larger at higher temperatures, but decreased faster and it was considerably smaller at the end of the experiment series as when carried out at lower temperatures. This effect was explained by the shielding effect of the large quantities of formed isoprene on the catalyst. The yield of isopentanes is larger at lower temperatures, because side-reactions do not occur at these temperatures. No isoprene forms at 500°. Therefore, it is preferable to repeat the regeneration of the catalysts when using it at higher temperatures (after 1 hour) and to carry out the reactions at high volume velocities (there is a possibility that more initial hydrocarbons are consumed). At 527° regeneration has to take place after 3 hours, and at 500° after 6-8 hours. There are 1 figure, 2 tables, and 3 Soviet references.

ASSOCIATION: Institut organicheskoy khimii im. N. D. Zelinskogo Akademii nauk SSSR (Institute of Organic Chemistry imeni N. D. Zelinskiy of the Academy of Sciences, USSR)

SUBMITTED: December 25, 1957
Card 2/2

SOV/79-29-2-18/71

AUTHORS:

Shuykin, N. I., Brusnikina, V. M.

TITLE:

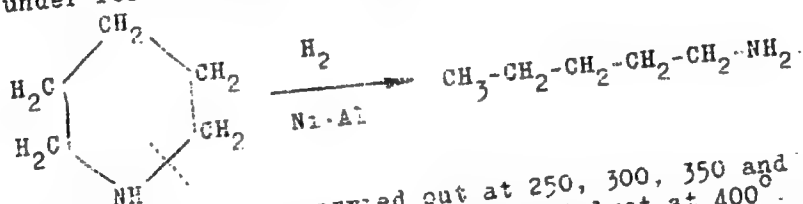
Catalytic Transformations of Piperidine in the Hydrogen Gaseous Envelope (Kataliticheskiye prevrashcheniya piperidina v atmosfere vodoroda)

PERIODICAL:

Zhurnal obshchey khimii, 1959, Vol 29, Nr 2, PP 438-441 (USSR)

ABSTRACT:

In the present paper the authors raised the question, whether it is possible for a hydrogenolysis of piperidine to take place in the current system over the skeleton nickel-aluminum catalyst under formation of -1-aminopentane, according to the scheme



This reaction was carried out at 250, 300, 350 and 400°, as well as by the use of copper-chromium catalyst at 400°. It was found that the destruction of the piperidine ring over the Ni-Al catalyst at increased temperature up to 400° chiefly takes place

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SOV/79-29-2-18/71

Catalytic Transformations of Piperidine in the Hydrogen Gaseous Envelope

under formation of a large amount of gaseous products, whereas hydrogenation over the copper chromium catalyst at the same temperature causes piperidine to dehydrogenate into pyridine. By the use of the former catalyst large quantities of α -picoline are formed in all cases the yield of which amounted up to 12.6% at the reaction temperature of 300°C. This circumstance allows the assumption that under these conditions methylene radicals appear on the surface of the catalyst, which transform the resulting pyridine into α -picoline (Scheme 2). There are 4 tables and 8 references, 4 of which are Soviet.

ASSOCIATION: Institut organicheskoy khimii Akademii nauk SSSR (Institute of Organic Chemistry of the Academy of Sciences, USSR)

SUBMITTED: December 2, 1957

Card 2/2

AUTHORS:

Shuykin, N. I., Bel'skiy, I. F.

SOV/79-29-2-19/7:

TITLE:

Hydrogenation of Furane Compounds Over the Ni-ZnO Catalyst.
(Gidrirovaniye furanovykh soyedineniy na Ni-ZnO-katalizatore)

PERIODICAL:

Zhurnal obshchey khimii, 1959, Vol 29, Nr 2, pp 442-445 (USSR)

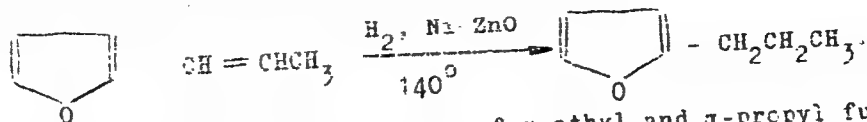
ABSTRACT:

Investigations carried out by Shuykin and other chemists (Refs 1-5) show that the properties of nickel catalysts are considerably dependent on the nature of the carrier. This is confirmed by the present investigation. The authors found that nickel stored upon ZnO hydrogenates the double bond in the side chain of α -alkenyl furane under conditions of hydrogenation in the vapor phase or, at higher temperatures, it allows hydrogenolysis of the furane cycle to take place only on the bond C-O, which is not close to the alkyl radical. This catalyst does not have the function of hydrogenating the double bonds in the furane cycle in the vapor phase. Thus, for example, silvane, that had been left over the above catalyst at 140°, was not transformed. Under the same conditions, α -propenyl furane was hydrated almost quantitatively into α -propyl furane.

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SOV/79-29-2.19/71

Hydrogenation of Furane Compounds Over the Ni-ZnO Catalyst



The hydrogenation of silvane, of α -ethyl and α -propyl furanes at 250° led to the complete cleavage of the cycle at the C-O bond 1-5 and, correspondingly, to the formation of pentanone-2, hexanone-3 and heptanone-4 (up to 95% yield) (Scheme 2). Thus, depending on temperature, the Ni-ZnO catalyst hydrogenates the olefin bond in the side chain of the alkenyl furanes in the selective way, or it may allow hydrogenolysis of the furane cycle at the C-O bond, which is not close to the alkyl radical. In the latter case, corresponding aliphatic ketones in yields up to 95% form at 250° . There are 1 table and 10 references, 5 of which are Soviet.

ASSOCIATION: Institut organicheskoy khimii Akademii nauk SSSR (Institute of Organic Chemistry of the Academy of Sciences, USSR)

SUBMITTED: November 28, 1957
Card 2/2

5 (3)
AUTHORS:

SOV/79-29-3-25/61
Shuykin, N. I., Bel'skiy, I. F., Savkina, O. N.

TITLE:

Hydrogenation of Silvan on Nickel Catalysts in the Liquid Phase
(Gidrirovaniye sil'vana na nikel'nykh katalizatorakh v zhid-
koy faze)

PERIODICAL:

Zhurnal obshchey khimii, 1959, Vol 29, Nr 3, pp 869-874 (USSR)

ABSTRACT:

In the present paper the authors investigated the hydrogenation capability of the skeleton-Ni-Al- and -Ni-ZnO-catalyst in the liquid phase to determine the influence of the phase conditions in carrying out the reaction upon the hydrogenation of the furan ring. The experimental data obtained were compared with the rules which were earlier found by the authors on hydrogenation of the furan homologues on the same catalysts in the vapor phase. These data led to the conclusion that the hydrogenation of the furan ring can proceed on one and the same catalyst in different directions, according to the reaction in the liquid or vapor phase. Silvan and α -propenyl furan were hydrogenated in the autoclave at 120-150° under a hydrogen pressure of 50-150 atmospheres excess pressure on the skeleton-Ni-Al-catalyst. The principal reaction products were, accord-

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SOV/79-29-3-25/61

Hydrogenation of Silvan on Nickel Catalysts in the Liquid Phase

ingly, tetrahydrosilvan and α -propyltetrahydrofuran (85-95%). On this hydrogenation of silvan in addition to tetrahydrosilvan (90%) the pentanediol-1,4 is formed, in which connection a certain part of the silvan molecule is subject to cleavage, forming H_2O and high-molecular compounds. Under the above reaction conditions, but on addition of water, about the same quantities of tetrahydrosilvan and pentanediol-1,4 are formed as well as about 7% of butanol-2 and pentanol-2 which are products of hydrogenolysis of the ring. The hydrogenolysis of the furan ring in silvan on hydrogenation on the skeleton-nickel-catalyst at 120-150° in the liquid phase proceeds similarly as in the vapor phase, i.e. on the C-O-bond 1-5 and on the bonds 1-5 and 4-5, yielding pentanol-2 and butanol-2, accordingly. The nickel on the ZnO is capable of hydrogenating the double bonds in the furan ring in the liquid phase at 200-250°, as contrasted with the vapor phase. Here, hydrogenolysis of the ring takes place only on the C-O-bond 1-5 yielding pentanone-2 as principal product which is not the case on the other catalysts. Thus the direction of the hydrogenation of the furan ring depends on the nature of the catalyst

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SOV/79-29-3-25/61

Hydrogenation of Silvan on Nickel Catalysts in the Liquid Phase

and on the phase conditions of the reaction. There are 1 table and 9 references, 7 of which are Soviet.

ASSOCIATION: Institut organicheskoy khimii Akademii nauk SSSR (Institute of Organic Chemistry of the Academy of Sciences, USSR)

SUBMITTED: December 20, 1957

Card 3/3

5 (3).

AUTHORS:

Shuykin, N. I., Bel'skiy, I. F.

SOV/79-29-3-26/61

TITLE:

Catalytic Reduction of the Phenyl Carbinols in Aromatic Hydrocarbons (Kataliticheskoye vosstanovleniye fenilkarbinolov v aromaticheskoye uglevodorody)

PERIODICAL:

Zhurnal obshchey khimii, 1959, Vol 29, Nr 3, pp 875-877 (USSR)

ABSTRACT:

If R in the compounds of the R-CH-^{OH}Alk type represents an

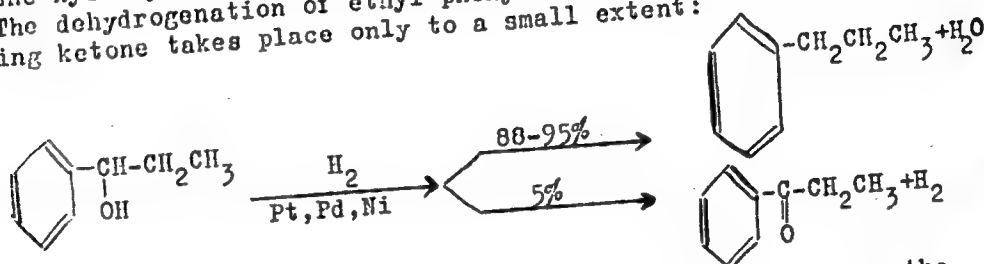
alkyl, no reduction of the hydroxyl group takes place on the catalysts Pd-C and Ni-ZnO, but more or less a dehydrogenation of the alcohol into the ketone. The question was of which chemical nature R should be, in order that an activated hydrogen could be substituted for the hydroxyl group by the catalytic method. The results of the investigations reported in the present paper show that with $R=C_6H_5$, using the catalysts Pt-C, Pd-C, Ni-ZnO and skeleton-Ni-Al, at 250° in the vapor phase a reduction of the hydroxyl group and a transformation of the phenyl carbinols into the aromatic hydrocarbons take place. Thus, e.g. ethyl phenyl carbinol is reduced at 250° in the vapor phase on all catalysts used to propyl benzene (yield 88-95%). On the Ni-Al-catalyst in addition to propyl

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SOV/79-29-3-26/61

Catalytic Reduction of the Phenyl Carbinols in Aromatic Hydrocarbons

benzil benzene (3%) and toluene (3%) are formed, owing to the hydrolysis process of the C-C-bonds in the side chains. The dehydrogenation of ethyl phenyl carbinol to the corresponding ketone takes place only to a small extent:



The catalytic reduction of benzene alcohol proceeds under the same conditions yielding toluene and dehydrogenation to benzaldehyde which is readily decarbonylated into benzene at 250°. The aliphatic alcohols (pentanol-2 and heptanol-1) are not reduced on the platinum and nickel catalyst, but, accordingly, dehydrogenated to give pentanone-2 and heptanal, with the latter being decarbonylated to n.-hexane (Scheme 2). There are 1 table and 1 Soviet reference.

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SOV/79-29-3-26/61
Catalytic Reduction of the Phenyl Carbinols in Aromatic Hydrocarbons

ASSOCIATION: Institut organicheskoy khimii Akademii nauk SSSR
(Institute of Organic Chemistry of the Academy of Sciences,
USSR)

SUBMITTED: December 20, 1957

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5(3)

SOV/79-29-4-10/77

AUTHORS:

Shuykin, N. I., Bel'skiy, I. F.

TITLE:

Hydrogenation of Furan Compounds on Platinum and Rhodium Catalysts (Gidrirovaniye furanovykh soyedineniy na platinovom i rodiyevom katalizatorakh)

PERIODICAL:

Zhurnal obshchey khimii, 1959, Vol 29, Nr 4, pp 1093-1096 (USSR)

ABSTRACT:

The hydrogenation results of furan compounds in the presence of catalysts containing metals of the group VIII of the periodic system lead to the conclusion that some of them are active predominantly in the hydrogenolysis of the double bond in the furan ring, and others develop a high activity in the hydrogenolysis of the ether bonds of the furan ring. As regards the action of the catalysts also the phases and temperatures play an important part in one and the same catalyst. Many examples are presented for this fact which underline that in general the comparison data on the properties of the catalysts can only be obtained under certain standard conditions during the reaction. For this reason the hydrogenation of silvan and α -vinyl furan on rhodium deposited on active charcoal in the vapor phase at 150, 200, and 300° was investigated. For comparison purposes also the hydro-

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SOV/79-29-4-10/77

Hydrogenation of Furan Compounds on the Platinum- and Rhodium Catalyst

genation of silvan and α -propenyl furan on platinated charcoal in the vapor phase at 150° was carried out. On the rhodium catalyst at 150° the furan ring hydrogenates very slowly which makes it possible to reduce the olefin bond in the side chain, maintaining the double bonds in the ring. α -vinyl furan is transformed at this temperature into α -ethyl furan (90% yield) (Scheme 1). The hydrogenation of silvan on Rh-C at 200° yields a mixture of tetrahydrosilvan (40%) and pentanone-2 (60%); at 300° only pentanone-2 is found in the hydrogenation products (Scheme 2). Similar results are offered by the hydrogenation products of both compounds on Pt-C at 150° . The Pd-C catalyst is found to be a specific catalyst of the hydrogenolysis of double bonds in the furan ring during the hydrogenation in the vapor phase ($140-275^{\circ}$). The Pt-C catalyst proved to be a specific catalyst of the hydrogenolysis of the furan ring on the C-O-bond 1-5 under the given conditions. The effect of the Rh-C catalyst is equal to that of the Pt-C catalyst at 300° ; at 200° it assumes an intermediary position between Pd-C and Pt-C as far as its properties are concerned. There are 1 table and 7 references, 6 of which are Soviet.

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SOV/79-29-4-10/77

Hydrogenation of Furan Compounds on the Platinum- and Rhodium Catalyst

ASSOCIATION: Institut organicheskoy khimii Akademii nauk SSSR (Institute of Organic Chemistry of the Academy of Sciences, USSR)

SUBMITTED: March 6, 1958

Card 3/3

5(3)

AUTHORS:

Shuykin, N. I., Bel'skiy, I. F.

SOV/79-29-4-11/77

TITLE:

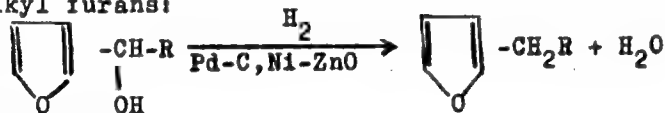
Catalytic Reduction of 2-Methyl-5-acetyl Furan
Into 2-Methyl-5-ethyl Furan (Kataliticheskoye
vosstanovleniye 2-metil-5-atsetilfurana v 2-metil-5-etilfuran)

PERIODICAL:

Zhurnal obshchey khimii, 1959, Vol 29, Nr 4, pp 1096-1099
(USSR)

ABSTRACT:

In addition to the reports of some chemists (Refs 1 - 6) on the part played by various catalysts under different conditions in the hydrogenation of furan ketones the authors investigated in the present paper the hydrogenation of 2-methyl-5-acetyl furan in the vapor phase at 250° on the catalysts Ni-ZnO, Ni-CdO and Pd-C. As was already earlier shown by the authors (Ref 7), on the hydrogenation of alkyl furyl carbinols on Pd-C and Ni-ZnO first the reduction of the hydroxyl group takes place, under forming the corresponding alkyl furans:



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Catalytic Reduction of 2-Methyl-5-acetyl Furan
Into 2-Methyl-5-ethyl Furan

SOV/79-29-4-11/77

As was to be expected, on the hydrogenation of the 2-methyl-5-acetyl furan the resulting methyl-(5-methylfuryl)-carbinol on Ni-ZnO and Ni-CdO in the vapor phase at 250° was subject to a further reduction into the 2-methyl-5-ethyl furan (Scheme 2), in contrast with the Pd-C catalyst nearly ineffective as regards the reduction of the carbonyl group and the hydrogenolysis of the furan ring into the 2-methyl-5-acetyl furan. As side reaction on the hydrogenation of the 2-methyl-5-acetyl furan on Ni-ZnO the hydrogenolysis of the ring takes place, thus yielding heptanone-2, which is scarcely the case on Ni-CdO (Scheme 3). The definite solution of this problem requires further investigations. The direct catalytic reduction of alkyl furyl ketones into alkyl furans may be of preparative importance since it is more convenient than the method of Kizhner (Ref 8) by way of the hydrazones, with subsequent reduction of these intermediate products. There are 9 references, 4 of which are Soviet.

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Catalytic Reduction of 2-Methyl-5-acetyl Furan
Into 2-Methyl-5-ethyl Furan

SOV/79-29-4-11/77

ASSOCIATION: Institut organicheskoy khimii Akademii nauk SSSR
(Institute of Organic Chemistry of the Academy of Sciences,
USSR)

SUBMITTED: March 6, 1958

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SOV/79-29-6-23/72

5(3)

AUTHORS:

Shuykin, N. I., Bel'skiy, I. F., Grushko, I. Ye.

TITLE:

On the Reaction of Tetrahydrofuran With Halogen Silanes (O vzai-
modeystvii tetragidrofurana s galoidsilanami)

PERIODICAL:

Zhurnal obshchey khimii, 1959, Vol 29, Nr 6,
pp 1882 - 1885 (USSR)

ABSTRACT:

From among all organic oxides those chemical transformations of the ethylene oxides are investigated most thoroughly, which yield the aliphatic compounds with reactive groups by the ready opening of the α -oxide ring under the influence of various agents. Far less investigated are the chemical transformations of the γ -oxides of tetrahydrofuran and its homologs. They possess a considerably lower reactivity and are of great importance for the organic synthesis since they can yield the 1,4-bifunctional derivatives of the aliphatic series: the dihalogen-alkanes, dinitriles, glycols, halogen-hydrins; besides the tetrahydrofuran derivatives are a sufficiently accessible and cheap material. In the present paper the reaction of tetrahydrofuran with alkyl- and aryl-chloro-silanes with two or three chlorine atoms in the molecule was investigated. It takes place only in the presence

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On the Reaction of Tetrahydrofuran With Halogen Silanes SOV/79-29-6-23/72

of anhydrous zinc (II) chloride in different directions according to the number of chlorine atoms in the chloro silane molecule. Methyl-dichloro-silane and dimethyl-dichloro-silane split up the tetrahydrofuran ring at both C-O bonds with 1,4-dichloro-butane being formed as main product (Scheme 1). In contrast to the dichloro-silanes the reaction of tetrahydrofuran with alkyl and aryl-trichloro-silanes takes place with ring opening only at one C-O-bond and yields the chlorine-substituted ester of the ortho-silicic acid (Scheme 2). In all cases the yields in mono-(δ -chloro-butoxy)-dichloro-silanes are very high (80-90%). Two chlorine atoms in the molecule of the mono (δ -chlorobutoxy)-dichloro-silane are active, but react mainly under formation of 1,4-dichloro-butane (Scheme 3). An interpretation of this reaction is suggested. The compounds synthesized are listed in two tables. There are 2 tables and 11 references, 1 of which is Soviet.

ASSOCIATION: Institut organicheskoy khimii Akademii nauk SSSR (Institute of Organic Chemistry of the Academy of Sciences, USSR)

SUBMITTED: April 18, 1958

Card 2/2

5(3)

AUTHORS:

Shuykin, N. I., Cherkashin, M. I.

SOV/79-29-7-26/83

TITLE:

On the Demethylation Reaction in the Hydrogenolysis of the Five-membered Cyclanes and n.-Alkanes on the Skeleton-Ni-Al-catalyst (O reaktsii demetilirovaniya pri gidrogenolize pyatichlennykh tsiklanov i n.-alkanov na skeletnom Ni-Al-katalizatore)

PERIODICAL:

Zhurnal obshchey Khimii, 1959, Vol 29, Nr 7, pp 2225-2230 (USSR)

ABSTRACT:

In earlier experiments made by several research workers (Refs 1-6) various methods with different nickel catalysts were applied to the hydrogenolysis of the C-C bonds of the five-membered cycle. These investigations showed that the catalytic properties of nickel catalysts are due to the nature of the carrier used. Thus, e.g. nickel on aluminum oxide (Refs 3, 5) exhibited a specific effect neither in five-membered cyclanes nor in alkanes of different structure, whereas nickel on deactivated kieselguhr demethylates isoalkanes selectively (Ref 6). It was therefore of interest to examine the effect of the skeleton nickel-aluminum catalyst frequently used in laboratories and industries also with respect to the

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On the Demethylation Reaction in the Hydrogenolysis SOV/79-29-7-26/83
of the Five-membered Cyclanes and n.-Alkanes on the Skeleton-Ni-Al-catalyst

hydrogenolysis of the C-C bonds of pentamethylene hydrocarbons and alkanes. This hydrogenolysis takes place under rather easy conditions and is accompanied by partial molecule simplification. Further experiments with this catalyst showed that at 200° and normal pressure it is capable of demethylating n.-alkanes. It was found that under the above conditions the molecule of dicyclopentyl is hydrogenolyzed only on one pentamethylene ring; in this connection also cyclanes with shortened chain are formed in addition to isoamyl cyclopentanes. There are 3 tables and 7 references, 6 of which are Soviet.

ASSOCIATION: Institut organicheskoy khimii Akademii nauk SSSR (Institute of Organic Chemistry of the Academy of Sciences, USSR)

SUBMITTED: June 9, 1958

Card 2/2

5(3)

AUTHORS:

SOV/79-25-7-27/83
Shuykin, N. I., Kononov, N. F., Kashkovskaya, L. K.

TITLE:

Catalytic Hydrodealkylation of Polyalkyl Benzenes
(Kataliticheskoye gidrodealkilirovaniye polialkilbenzolev).
I. Demethylation of Toluene Over 10% Ni-Al₂O₃. The Effect of
Hydrogen Pressure (I. Demetilirovaniye toluola na 10% Ni-Al₂O₃.
Vliyaniye davleniya vodoroda)

PERIODICAL:

Zhurnal obshchey khimii, 1959, Vol 29, Nr 7, pp 2230-2235 (USSR)

ABSTRACT:

Toluene, which, compared to the important benzene, was industrially only little utilized, has frequently been investigated (Refs 1-12) for the purpose of converting it into benzene by catalytic methods. Neither in the papers mentioned nor in patents attention was paid to the stability of the catalysts since in most cases the maximum duration of the experiments was 3 hours. The present paper dealt with the investigation of the selective conversion of toluene into benzene over an active and sufficiently stable catalyst. In this case the hydrogenation reactions of the benzene nucleus, the regrouping of the methyl groups, the hydrocracking process,

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Catalytic Hydrodealkylation of Polyalkyl Benzenes.

SOV/79-29-7-27/83

I. Demethylation of Toluene Over 10%-Ni-Al₂O₃. The Effect of Hydrogen Pressure

and the thermal decomposition under the formation of coke should take place only to a small degree. As is known, an extensive cleavage of toluene takes place at normal hydrogen pressure and approximately 450° with an impurification of the platinum-, nickel-, and other catalysts by coke; at increased pressure, on the other hand, the undesired hydrogenation of the benzene nucleus takes place. Therefore, the influence of hydrogen pressure on the demethylation of toluene had to be investigated in the first place. The experiments and the apparatus used are described in the experimental part. The demethylation of toluene into benzene took place at a pressure of 5 at without formation of hydroaromatic hydrocarbons, without a regrouping of the methyl groups, and without hydrocracking of benzene to methane. The yield in the catalyzate (with a benzene content of 30% approximately) is approximately 85% , computed for toluene. At a considerably higher hydrogen pressure (25 atmospheres excess pressure) and under otherwise equal conditions considerable amounts of hydroaromatic hydrocarbons are formed (cyclo- and methyl cyclohexane).

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Catalytic Hydrodealkylation of Polyalkyl Benzenes. SOV/79-29-7-27/83
I. Demethylation of Toluene Over 10% Ni-Al₂O₃. The Effect of Hydrogen Pressure

In this case also toluene was subjected to a hydrocracking process. There are 2 figures, 2 tables, and 14 references, 9 of which are Soviet.

ASSOCIATION: Institut organicheskoy khimii Akademii nauk SSSR (Institute of Organic Chemistry of the Academy of Sciences, USSR)

SUBMITTED: June 14, 1958

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5(3)

SOV/79-29-9-35/76

AUTHORS: Shuykin, N. I., Bel'skiy, I. F.

TITLE: On the Reaction of Propylene Oxide With Some Compounds Containing an Active Chlorine Atom

PERIODICAL: Zhurnal obshchey khimii, 1959, Vol 29, Nr 9, pp 2973-2975 (USSR)

ABSTRACT: The present paper describes the investigation of the reaction of propylene oxide with $AlCl_3$, $TiCl_4$, and PCl_3 . The two first chlorides were previously (Ref 9) used in the reaction with tetrahydrosilane so that it was possible to compare their effects upon α - and γ -oxides. The reaction products of propylene oxide with $AlCl_3$, $TiCl_4$, and PCl_3 decomposed in distillation already at reduced pressure; for this reason they were subjected to hydrolysis. An analysis of the products yielded the following results: 1) The reaction with $AlCl_3$ proceeds in a similar way as the reaction of tetrahydrosilane (Ref 9), i.e. under the formation of 1,2-dichloro propane (Scheme 2). The products of hydrolysis do not contain chlorohydrin. 2) PCl_3 and $TiCl_4$ open propylene oxide on the C-O-bond next to the methyl

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group, which leads to unstable chlorine-substituted esters of phosphorous- and titanous acid. Hydrolysis of these esters yields 70-85% 2-chloropropanol-1 (Scheme 3). In order to prove the structure of the final product (chlorohydrin), the latter was transformed into the acetate which by reducing dehalogenation in the vapor phase over Pt-C at 300° could be smoothly quantitatively transformed into propyl acetate without difficulty (Scheme 4). This method of proving the structure of halohydrin is more convenient than the usual oxidation method. Titanium tetrachloride is known to cause a splitting of the γ -oxide cycle under elimination of the oxygen atom and formation of 1,4-dichloropentane in the reaction with tetrahydrosilane. PCl_3 and TiCl_4 therefore have the same effect upon the α -oxide cycle as the other acid chlorides of inorganic acids, e.g. AsCl_3 , SiCl_4 , etc. There are 11 references, 8 of which are Soviet.

ASSOCIATION: Institut organicheskoy khimii Akademii nauk SSSR (Institute of Organic Chemistry of the Academy of Sciences, USSR)

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5(3)

SOV/79-29-8-29/8:

AUTHORS: Shuykin, N. I., Bel'skiy, I. F., Grushko, I. Ye.

TITLE: On the Reaction of Tetrahydrosilane With Silicon Tetrachloride

PERIODICAL: Zhurnal obshchey khimii, 1959, Vol 29, Nr 8,
pp 2591 - 2594 (USSR)

ABSTRACT: In the present paper, the authors investigated the reaction of tetrahydrosilane with silicon tetrachloride. This reaction takes place only in the presence of anhydrous zinc chloride. The reaction products underwent an intense decomposition in the vacuum distillation. For this reason, they were previously subjected to a hydrolysis with water. The products of this hydrolysis had to be fractionated at reduced pressure with a column top section. In this connection, the 1,4-dichloropentane (15-20%) and the chlorine-substituted amyl alcohol (70-80%) were separated, the latter of which was likewise obtained on hydrolysis of the chlorine esters of silicic acid (Scheme 1). Depending on the cleavage of the C-O bond in position 1-2 or 1-5, the alcohol can be formed with a primarily (I) or secondarily (II) bound hydroxyl group (4-chloro-pen-

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Tetrachloride

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tanol-1 or 5-chloro-pentanol-2). The structure of the chlorohydrin obtained by hydrolysis was confirmed according to the transformations of scheme 2. The reducing dehalogenation of 2-chloro-5-acetoxy-pentane (III) on platinized carbon in the vapor phase proceeds smoothly and with high yields (95%) at the primary amyl acetate (IV). The chlorohydrin obtained by hydrolysis of the reaction products of tetrahydrosylvane with SiCl_4 thus represents the 4-chloro-pentanol-1. This means that the ring in the tetrahydrosylvane is cleft at the C-O bond adjoining the methyl group, under the influence of SiCl_4 . There are 10 references, 4 of which are Soviet.

ASSOCIATION: Institut organicheskoy khimii Akademii nauk SSSR (Institute of Organic Chemistry of the Academy of Sciences, USSR)

SUBMITTED: July 4, 1958

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3(5)

SOV/79-29-9-34/76

AUTHORS:

Shuykin, N. I., Bel'skiy, I. F., Minachev, Kh. M.

TITLE:

Hydrogenation of the Furan Compounds by Means of Metals.
VIII. Groups of the Periodic System

PERIODICAL:

Zhurnal obshchey khimii, 1959, Vol 29, Nr 9, pp 2969-2973 (USSR)

ABSTRACT:

Owing to the results of previous investigations (Refs 1-5) the problem arose which of the metals of group VIII are like palladium, capable of bringing about the catalytic hydrogenation of the furan cycle in a wide temperature range, and which of them are the most active ones. The catalytic properties of osmium, iridium, and ruthenium, applied to carbon, were investigated in the hydrogenation of silvane and α -vinyl furan in the vapor phase and at various temperatures. α -Vinyl furan is hydrogenated on all these catalysts at 150° into α -ethyl furan in yields of 95-100% (Scheme 1). On Ir-C and Ru-C a small portion (~5%) of α -ethyl furan formed is subjected to hydrogenolysis on the C-O bond 1-5 (Scheme 2). Hydrogenation of silvane on Os-C, Ir-C, Ru-C at higher temperatures shows that these catalysts are not able to reduce the furan cycle, but are only capable of hydrogenolysis on the C-O bond 1-5 under the formation of the

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Hydrogenation of the Furan Compounds by Means of Metals. VIII. Groups of the Periodic System

methylpropyl ketone (Scheme 3). The results of this paper as well as those already previously obtained permit to draw some general conclusions. All catalysts containing metals of group VIII, applied to carbon could be in hydrogenation conditionally divided in the vapor phase under comparable temperature conditions into two groups, depending on their effect upon the furan cycle: 1) The catalysts of the platinum type (Pt, Os, Ir, Ru, Rh) show either a weak or practically no capability of hydrogenation of the double bond in the furan cycle. On these catalysts only hydrogenolysis of the furan cycle on the C-O bond 1-5 occurs at 200-300°. 2) The catalysts of the palladium type are in a sufficiently wide temperature range capable of hydrogenating the double bond in the furan cycle. Hydrogenolysis of this cycle occurs only at higher temperatures. There are 7 Soviet references.

ASSOCIATION: Institut organicheskoy khimii Akademii nauk SSSR (Institute of Organic Chemistry of the Academy of Sciences, USSR)

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5(3)

AUTHORS:

Shuykin, N. I., Corresponding
Member AS USSR, Bel'skiy, I. F.

SOV/20-125-2-29/64

TITLE:

Hydrogenation of the Furan Compounds on Skeleton Catalysts
(Gidrirovaniye furanovykh soyedineniy na skeletnykh
katalizatorakh)

PERIODICAL:

Doklady Akademii nauk SSSR, 1959, Vol 125, Nr 2, pp 345-347
(USSR)

ABSTRACT:

The Ni-Al catalyst is the best-explored among the catalysts mentioned in the title (Refs 1-3). Unlike this catalyst, the Ni-Zn-skeleton catalyst shows very little activity with regard to the hydrogenization of the furan cycle in the vapor phase. At 150° and normal pressure it is effected preponderantly, at 250-300°, however, exclusively, at the C-O bond which does not border on the lateral alkyl group (α -position). The skeleton copper catalyst (copper-aluminum alloy) was studied in the hydration reaction of furfural in the liquid and vapor phases. In the former phase, furfural was quantitatively reduced to furfuryl alcohol at 140° and 100 atmospheres excess pressure. Thus copper in this case behaves like copper chromite, although its activity is noticeably lower. On the

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Cu-Al catalyst, at normal pressure and below 200°, furyl alcohol, and at 250° silvan were formed as main products in the vapor phase (Ref 5). Temperature increase favors the hydrogenolysis of the cycle (Ref 6). The authors investigated the catalytical properties of skeleton platinum, palladium, and copper. These substances were manufactured by means of a partial removal of aluminium from the alloys of these metals with aluminium. It is a well-known fact that Pt and Pd, deposited on coal, show markedly different properties as far as their relation to the hydration and hydrogenolysis reactions of the furan cycle in the vapor phase are concerned. The results of the investigations under consideration show that the skeleton Pt-Al and Pd-Al catalysts, in their properties, are analogous to platinum and palladium, if the latter are deposited on coal. Thus skeleton Pd-Al, at 150° in the vapor phase, readily hydrates the furan cycle by transforming for example silvan and 2-methyl-5-ethyl-furan into the corresponding tetrahydrofurans. The hydrogenolysis of the furan cycle becomes noticeable only at comparatively high temperatures. The hydrogenolysis degree of the furan ring

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at 270° is identical on both Pd-C and Pd-Al, and amounts to 25%. The analogy holds true also for the hydration of isopropyl-furyl-carbinol at 250°. The multiple bonds of the furan ring are hydrated only after the reduction of the hydroxyl group. From this it will be clear that the selectivity in the reduction of the hydroxyl group depends on the speed of the passage over the catalyst of the alkyl-furyl-carbinol. Thus at normal pressure the skeleton Pt-Al and Cu-Al catalysts can according to temperature conditions either hydrate the double bonds in the side chain or else bring about the hydrogenolysis of the furan cycle. There are 2 tables and 8 references, 4 of which are Soviet.

ASSOCIATION: Institut organicheskoy khimii im. N. D. Zelinskogo
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imeni N. D. Zelinskiy of the Academy of Sciences,
USSR)

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5(3)

AUTHORS:

Shuykin, N. I., Corresponding Member, SOV/20-125-5-27/61
AS USSR, Bel'skiy, I. F., Karakhanov, R. A.

TITLE:

Thermal Isomerization of 2-Methyl-4.5-dihydrofuran to
Methyl-cyclopropyl Ketone (Termicheskaya izomerizatsiya
2-metil-4,5 digidrofurana v metiltsiklopropilketon)

PERIODICAL:

Doklady Akademii nauk SSSR, 1959, Vol 125, Nr 5,
pp 1051-1052 (USSR)

ABSTRACT:

According to reference 1 2.3-dihydrofuran isomerizes at 375°
to cyclopropane aldehyde. A small quantity of croton aldehyde
is formed therein, which is partially decarbonylated to
propylene under the experimental conditions. The gas
development increases with rising temperature. The first
reaction is reversible. The inequivalence of the C-O bonds
depends in the dihydrofuran molecule only on the presence
of the double bond. This very fact is bound to determine
the place of rupture of the dihydrofuran ring. Wilson (Ref 1)
assumed that 2.3-dihydrofuran behaves in the aforesaid
isomerization like simple vinyl ethers. According to
formal-structural considerations it is, however, clear that

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Thermal Isomerization of 2-Methyl-4.5-dihydrofuran to SOV/20-125-5-27/61
Methyl-cyclopropyl Ketone

the formation of cyclopropane aldehyde in the rupture of the dihydrofuran ring is possible at the C-O bond 1.2 as well as at the C-O bond 1.5. Therefore the results of the isomerization of the 2.3-dihydrofuran cannot indicate definitely the place of rupture of the dihydrofuran ring. In the case of the isomerization of α -alkyl-dihydrofurans two different products - aldehyde or ketone - are bound to be produced according to the C-O bond which has been ruptured. The authors tried to investigate the isomerization of such α -alkyl-substituted 2.3-dihydrofurans mentioned in the title, the alkyl group of which is found at the double bond. They found that 2-methyl-4.5-dihydrofuran isomerizes to methyl-cyclopropyl ketone at 475 - 500° (Reaction II, see Scheme). Thus, the C-O bond 1.5 is ruptured and a new bond is formed between the C-atoms 3 and 5 and the keto-group in the side chain. The reaction carried out here is irreversible, in contrast with the isomerization of 2.3-dihydrofuran to cyclopropane aldehyde which is reversible. There are 2 references.

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